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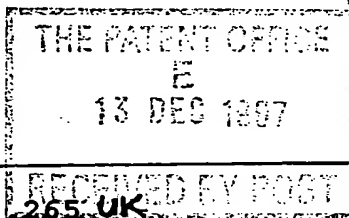
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Cardiff Road  
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57 265 UK

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13 DEC 1997

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3. Full name, address and postcode of the or of each applicant (underline all surnames)

Sofitech N.V. (SOFITECH)  
Rue de Stalle 140  
B-1180 Brussels  
Belgium

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

Belgium

06546303002

4. Title of the invention

Stabilizing clayey formations

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

MIRZA, Akram Karim  
c/o Schlumberger Cambridge Research Limited  
High Cross  
Maddingley Road  
Cambridge CB3 0EL

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Date of filing  
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Akram Mirza  
01223 325447

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## STABILIZING CLAYEY FORMATIONS

This invention relates to compositions and methods for stabilizing subterranean clayey formations surrounding a borehole. More specifically, it pertains to clay stabilizing additives for aqueous fluids used in drilling, completing and maintaining boreholes.

## BACKGROUND OF THE INVENTION

When geological formations containing water swelling clays come in contact with water, particularly fresh water, clays in the formations may swell and/or disperse with attendant loss of permeability and/or mechanical strength to interfere with recovery of petroleum or other minerals from the formations. Swelling and dispersion occur when aqueous fluids used in oil recovery come in contact with the clays. Clayey formations are often impermeable or have low permeability or lose part or all of their permeability on contact of the clays with water or water base systems such as injection fluids, drilling muds, stimulation fluids and gels. Dispersed clays may also invade a permeable producing formation during drilling to create low permeability zone in the vicinity of the borehole.

Given the importance and the ubiquity of clayey or shaley formations, it is not surprising that much effort has been put into developing and improving additives for clay or shale inhibition. Various methods and additives can be found for example in the United States Patent Nos. 5342530, 5211250, 5197544, 5152906, 5099923, 5097904, 5089151, 4842073, 4830765, 4828726, 4563292, 4536303, 4536304, 4536305, 4505833, 4497596, 4172800 and 3578781.

These additives usually are salts and/or polymers which effectively prevent the water from permeating the clay.

Furthermore, it is known to consolidate sandstone and other highly porous and weak formations with a fluid containing

polymerizable materials, such as resins or isocyanates in combination with diols. Those methods are described for example in the United States Patent Nos. 5242021, 5201612, 4965292, 4761099, 4715746, 4703800, 4137971, or 3941191. It is however  
5 important to note that the porosities of sandstones and similar formations differ from those of shale formations by several orders of magnitude. The consolidation of highly porous, unstable sandy formations and shale formations with a very low porosity are therefore generally recognized in the art as  
10 separate technical fields.

In technical fields unrelated to the present invention, efforts to form composites of clayey materials have been described. The known methods of forming so-called "nanocomposites" include the  
15 addition of a reactive (monomeric or polymeric) species to clays which have been previously treated with another compound with which it will react. The ensuing chemical reaction can occur in one of two ways: either the second additive is capable of physically cross-linking the polymer, or it promotes further  
20 self-polymerization. Such processes can result in nanocomposite silicate-polymers which attain a certain degree of stiffness, strength and barrier properties with far less ceramic content than comparable glass- or mineral-reinforced polymers. As such they are far lighter in weight than conventionally filled  
25 polymers.

Examples are provided by the following references: 'Polyamide-Organoclay Composites', S. Fujiwara and T. Sakamota, Japan, Patent 51 109,998, 1976; 'Composite Material Containing a  
30 Layered Silicate', A. Usaki et al, Toyota, U.S., U.S. Patent 4, 889, 885, (1989); M.S. Wang and T.J. Pinnavaia, 'Clay-Polymer Nanocomposites Formed from Acidic Derivatives of Montmorillonite and an Epoxy Resin', *Chem. Mater.*, **6**, 468, (1994); T.J. Pinnavaia et al, 'On the Nature of Polyimide-Clay Hybrid  
35 Composites', *Chem. Mater.*, **6**, 573, (1994); P.B. Messersmith and E.P. Giannelis, 'Synthesis and Characterization of Layered Silicate-Epoxy Nanocomposites', *Chem. Mater.*, **6**, 1719, (1994); T. Lan and T.J. Pinnavaia, 'Clay-Reinforced Epoxy

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Nanocomposites, *Chem. Mater.*, **6**, 2216, (1994); E.P. Giannelis, 'Polymer Layered Silicate Nanocomposites', *Adv. Mater.*, **8**, 29, (1996); T.J. Pinnavaia et al, 'Epoxy Self-Polymerization in Smectite Clays', *J. Phys. Chem. Solids*, **57**, 1005, 1996. In spite  
5 of the stabilization and strengthening that these additives impart to the resultant nanocomposite materials, there are currently several limitations to this technology which are important from an oilfield perspective. One is that the established methodology necessarily involves a high-temperature  
10 curing process for the chemical reactions to take place; another is that such an approach inevitably results in the production of composites in which the silicate is delaminated and randomly distributed within the polymer matrix. No technology has been developed which is capable of stabilizing clay silicates under  
15 ambient, aqueous conditions.

In view of the above, it is an object of the invention to provide a novel method of stabilizing subterranean clayey formations surrounding a borehole. It is another, more specific  
20 object of the invention to provide clay stabilizing additives for aqueous fluids used in drilling, completing and servicing boreholes under ambient conditions.

25

## SUMMARY OF THE INVENTION

This invention is concerned with the identification of a wide range of compounds which are capable of strengthening and stabilizing clay and shale minerals through a process of in-situ  
30 polymerization. These diverse compounds (or "additives") include both monomers and polymers, in aqueous solution or suspension. The resulting nanocomposite materials, which have dramatically enhanced mechanical properties compared with the original clay and shale samples, have a vast range of potential materials-  
35 science and materials-technology applications both within and outside the oilfield.

In chemical terms, the invention comprises a wide variety of additives which are capable of stabilizing clay films in aqueous solution. The additives that have been tested encompass four broad areas: (1) species which are capable of intercalating clay galleries and affording stabilization such as diamines, polyethylene glycols (PEGs), polypropylene glycols (PPGs) and polymeric diamines; (2) reagents which are capable of undergoing condensation reactions and thus polymerizing in-situ such as diamines, aldehydes, ketones, dicarboxylic acids; (3) reagents which are capable of ring opening of epoxides or acrylates effecting polymerization in-situ such as alcohols, amines; (4) reagents which are capable of self-polymerization within clay galleries such as alkenes. All of these techniques provide considerable stabilization over untreated films.

There are three main applications of the invention. Firstly, it is envisaged that a drilling mud formulation containing a combination of the specified compounds described below may be used as a clay and shale swelling inhibitor fluid, to maintain the integrity of the wellbore during conventional drilling operations. Secondly, a completion fluid formulation containing a combination of the same compounds may be used for general remedial operations in the wellbore. Finally, the invention may be used to achieve the goal of "casingless drilling", that is to achieve with one and the same drilling and completion fluid the equivalent result of what is today obtained through a combination of drilling, casing and cementing operations.

These and other features of the invention, preferred embodiments and variants thereof, and further advantages of the invention will become appreciated and understood by those skilled in the art from the detailed description and drawings following below.

#### EXAMPLE(S) FOR CARRYING OUT THE INVENTION

The level of clay stabilization provided by different additives and formulations were assessed by a laboratory technique.



In the testing procedure a small piece of montmorillonite clay film is treated with a combination of reagents which might be capable of cross-linking or polymerizing inside clay layers and  
5 examining the resulting clay for strength and rigidity in a qualitative manner.

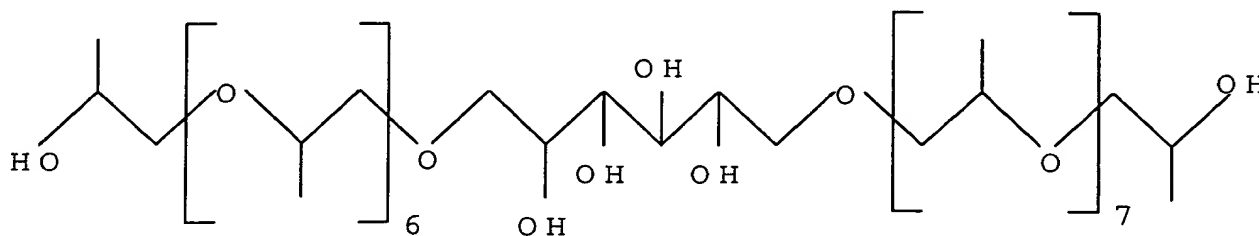
All the film tests described below were performed on the same batch of montmorillonite clay films. The films have been stored  
10 in a refrigerator in sealed Petri dishes and checked periodically by infrared spectroscopy to ensure integrity of the samples.

A very wide range of potential stabilizers were examined in  
15 terms of their chemical reactivity, structure and the conditions under which the tests were performed. All tests have been performed in an identical manner by the addition of a piece of montmorillonite film to an aqueous solution of the intercalating agent (5cm<sup>3</sup> of a 5% w/w solution), the pH of which had already  
20 been adjusted (where necessary, with 10% v/v aqueous HCl solution). The potential cross-linking reagent was then immediately added. Films were then allowed to stand in the reaction mixture for one day before washing with distilled water and were then placed in fresh distilled water to monitor their  
25 long term stability.

The reagents investigated are abbreviated in the following (including the Appendix 1) as: A = acrolein; AA = adipic acid; BA = boric acid; DAP = 1,5-diaminopentane; EDA = ethylene-1,2-  
30 diamine or 1,2-diaminoethane; EPP = 1,2-epoxy-3-phenoxypropane; FS = 37 %w/v formaldehyde solution; GA = 50% w/v glutaric aldehyde solution; MA = methyl acrylate; OA = oxalic acid; PA = 2-acetylpyridine; PC = 2-pyridinecarboxaldehyde; PO = propylene oxide; PEDG = polyethylenediglucamide; PPDG =  
35 polypropylenediglucamide.

The intercalating reagents investigated included  $\text{CH}_3\text{CH}(\text{NH}_2)\text{CH}_2\text{-}[\text{OCH}(\text{CH}_3)\text{CH}_2]_1\text{-}[\text{OCH}_2\text{CH}_2]_m\text{-}[\text{OCH}_2\text{CH}(\text{CH}_3)]_n\text{-NH}_2$  (BNH<sub>2</sub>), DAP, EDA, PA,

PC, PEDG, PPDG and a reaction product of glycol and ethylenoxide with the structural formula



5 denoted as PEG in Appendix 1. And the potential cross-linkers were A, AA, BA, EPP, FS, GA, MA, OA, PPDGE, PO and styrene. Cross-linkers were added as either neat reagents (0.5cm<sup>3</sup> A, EPP, MA, PPDGE, PO and styrene and 0.5g BA) or as aqueous solutions (5cm<sup>3</sup> 5%w/w solution AA, OA; 1cm<sup>3</sup> of 50% w/w solution GA and 5cm<sup>3</sup> 10 of a 37% w/v solution FS).

The montmorillonite film tests provided a means of ascertaining the potential of a number of reagent combinations; the results are summarized in Appendix 1. (In the table in Appendix 1, the 15 term "Stable" applied to described the film stability means that such films do not dissolve in water, while "Exfoliates" simply means they are visibly fatter after treatment. The latter term does not mean the films fall apart; this only happens if extreme exfoliation occurs and the resulting material does not have a 20 matrix around it which "sets".) These combinations can be broadly divided into four categories, i.e., (1) Substrate intercalation; (2) Condensation stabilization with no pH adjustment; (3) Stabilization through epoxide ring opening under neutral or acidic conditions; (4) Substrate in-situ 25 polymerization.

#### (1) Substrate Intercalation

Three films (3, 6 and 24 according to the numbering system used 30 in Appendix 1) were treated with potential intercalators without any chemical cross-linking reagent capable of forming covalent bonds. It was found that, by mixing PEG and BNH<sub>2</sub> solutions in equal quantities, a very stable film could be produced. This is

consistent with the effect of  $\text{BNH}_2$  alone but shows considerable improvement on the stabilization afforded by PEG, indicating that amines have superior clays stabilization properties.

Further attempts were made to stabilize the PEG films by

- 5 addition of boric acid (film 6) as a potential cross-linking template for the hydroxyl groups of the polymer and those of the clay galleries, but no improved stability was observed as compared with PEG alone.

#### 10 (2) Condensation Stabilization

These films are generally the most robust films that have been produced (1, 7, 23, 34-43), with particularly impressive properties being associated with films 7, 34, 37, 40, 42 and 43.

- 15 The equilibrium between reactants and products for this system under aqueous conditions clearly favors the reactants. It is therefore proposed that upon intercalation in the interlamellar layer an anhydrous environment is created through the concomitant expulsion of water. These conditions facilitate the
- 20 condensation process and lead to film stabilization whilst preventing substrate polymerization in solution.

#### (3) Ring Opening Stabilization

- 25 The chemical process resulting from the ring opening of epoxides is akin to that involved in the industrial synthesis of a number of polymers including PEG--a chain reaction is started after initial ring opening, resulting in an in-situ epoxide polymerization. To produce a stable film acidic solutions must
- 30 be employed, the acidity of which is dependent on both the intercalator and the cross-linker. When the diamines EDA, DAP and  $\text{BNH}_2$  are used only mildly acidic (ca pH 6) conditions are required with PO and EPP to produce very robust films (10, 11, 14, 15 and 25-27). Curiously, with PPDGE much more acidic
- 35 conditions (pH 2) produce more impressive films (28, 29) than at less acidic pH (films 9, 29). The exfoliation observed in film 13 at pH 6 is not observed in film 29 despite conditions which should favor more rapid reactions. Stabilization of PEG treated

films is more difficult. If the same mildly acidic conditions are employed as were successful for the diamines, the treated films are highly exfoliated and eventually decompose (films 18, 19, 21, 22). Stable films can be produced by adding a small  
5 quantity of a diamine solution to the bulk PEG liquor and treating them with epoxides under mildly acidic conditions (16B and 17). Films can also be stabilized with PEG and all of the epoxides but very severe conditions (pH 2) are required (30-32); the resultant films are very soft although they are stable.

10

#### (4) Substrate in-situ Polymerization

Of the four methods adopted to produce stable films, this method has been the least successful. The polymerization of a substrate  
15 necessitates a reaction initiator and it is difficult to control this process. This problem is highlighted by film 12 which resulted in the entire test solution polymerizing. Generally this reaction type produces exfoliated films (2, 4, 5, 8 and 20) which are often considerably swollen and blistered, indicating  
20 that the reactions are rapid and too potent for the films. Clearly a solution which polymerizes as is the case for 12 is undesirable but the stable film produced with EDA and A (film 33) indicates that there may be future applications to this methodology.

25

Evidence for absorption into the interlamellar layer of substrates

It is important to establish that the reagents are actually  
30 binding within the interlamellar layers of the films rather than merely polymerizing on the film surface. Evidence for an intercalation effect was established through the use of X-ray diffraction studies on a number of stable films; see Appendix 1. It can be clearly seen that changing the intercalating substrate  
35 changes the d-spacings of the montmorillonite films. What perhaps provides more conclusive evidence of a genuine polymerization within the interlamellar layer are the range of d-spacings that are observed for the intercalating substrate,

EDA. Values ranging from 12.72 (film 36) to 17.72 (film 34) are observed. Both of these films are stable, flexible, hard and show no signs of exfoliation typical of swelling due to water viz. marked whitening. Perhaps even more revealing are the different spacings observed for the same substrates in films 36-38. Although the treated films all have relatively similar properties (with perhaps most superior behavior attributable to film 37) the d-spacings are remarkably different. The two films (36 and 38) which have been adjusted to acidic pH both show similar d-spacings of ca. 13Å whereas film 37 (in which unchanged substrates have been employed) shows a d-spacing of ca. 15Å.

#### Stability of Treated Films

All stable films were washed thoroughly with water and then placed in distilled water to investigate their long term stability under saturated aqueous conditions. All of the treated films (1, 7, 10, 14, 16B, 17, 25, 27-43) show great stability with only films 31 and 32 showing any signs of decomposition after up to four months. These two films do showed some exfoliation. One interesting effect is observed after prolonged soaking of films treated with EPP. All, with the exception of film 25, become coated with a sticky layer and some (10, 14, 17) become transparent. The sticky coating can be accounted for due to epoxide leaching from the clay and subsequent polymerization on its surface. The second effect may relate to a change in the refractive index of the film in some way upon coating with the polymerizing epoxide. This effect also substantiates the absorption of substrates into the clay films (see also the X-ray diffraction results) as all substrates external to the clay are removed on rinsing. It is worthwhile noting that although the physical properties of these films have changed over a period of time, their d-spacings do not vary markedly. The d-spacings of films 1, 7, 10, 14, 16B and 17, after four months soaking in water, are 13.97Å, 14.04Å 14.96Å, 17.17Å, 17.20Å and 17.87Å respectively. The biggest difference between previous measurements and the post-soaking results lies in film 10 which shows a change of 0.583Å. The other numerical data are

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considered to be within the experimental errors of the measurement.

Appendix 1. Table summarizing the properties of clay nanocomposite materials synthesized

Film	Intercalator	Cross-linker	pH	d-spacing (Å)	Film Stability		Film Condition		
					Exfoliates	Stable	Brittle	Flexible	Hard or Soft
1	BNH <sub>2</sub>	FS	na	14.03		Yes		Yes	Hard
2	BNH <sub>2</sub>	MA/H <sub>2</sub> O <sub>2</sub>	na	-	Yes		Yes		Hard
3	BNH <sub>2</sub> /PEG	-	na	-		Yes	Yes		Hard
4	PEG	MA	na	-	Yes				Soft
5	PEG	MA/H <sub>2</sub> O <sub>2</sub>	na	-	Yes				Soft
6	PEG	BA	na	-	Yes				
7	EDA	FS	na	14.43		Yes		Very	Hard
8	FS	Catalytic H <sub>2</sub> O <sub>2</sub>	na	-	Yes	Yes	Yes		Hard
9	EDA	PPDGE	6	-		Yes	Yes	Some	
10	EDA	EPP	6	15.55		Yes		Yes	Hard
11	EDA	PO	6	-		Yes		Yes	
12*	EDA	A	na	-					
13	BNH <sub>2</sub>	PPDGE	6	-	Yes	Yes	Yes		Hard
14	BNH <sub>2</sub>	EPP	6.4	-		Yes		Yes	Hard
15	BNH <sub>2</sub>	PO	6.6	-		Yes	Yes	Some	Hard
16B	4PEG:1BNH <sub>2</sub>	EPP	4	17.31		Yes		Yes	Hard
17	4PEG:1EDA	EPP	5	17.66		Yes	Slightly	Yes	Hard
18	PEG	EPP	na	-	Yes				Soft
19	PEG	PO	na	-	Yes				Soft
20	-	Styrene/H <sub>2</sub> O <sub>2</sub>	na	-	Yes				Soft
21	PEG	EPP/H <sub>2</sub> O <sub>2</sub>	na	-	Yes				Soft
22	PEG	PO/H <sub>2</sub> O <sub>2</sub>	na	-					Soft
23	DAP	FS	na	-		Yes	Yes		Hard
24	DAP	-	na	-		Yes		Yes	Hard
25	DAP	EPP	6.5	18.55		Yes	Slightly		Hard
26	DAP	PO	6.5	-		Yes	Yes		
27	DAP	PO/EPP	na	17.15		Yes	Yes		Hard
28	EDA	PPDGE	2	14.14		Yes		Yes	Hard

Appendix 1 (continued)

Film	Intercalator	Cross-linker	pH	d-spacing (Å)	Film Stability		Film Condition		
					Exfoliates	Stable	Brittle	Flexible	Hard or Soft
29	BNH <sub>2</sub>	PPDGE	2	15.90		Yes	Yes		Hard
30	PEG	PPDGE	2	17.51		Yes		Yes	Soft
31	PEG	PO	2	16.54	Yes	Yes			Soft
32	PEG	EPP	2	16.83	Yes	Yes			Soft
33	EDA	A	na	15.07		Yes		Yes	Hard
34	EDA	PC	na	17.72		Yes		Yes	Hard
35	EDA	PA	na	15.06		Yes			Hard
36*	EDA	AA	6	12.72		Yes		Yes	Hard
37	EDA	AA	na	14.98		Yes		Very	Hard
38*	EDA	AA	2	13.00		Yes		Yes	Hard
39*	EDA	OA	6	15.47		Yes	Yes	Slightly	Hard
40	EDA	OA	na	15.06		Yes		Very	Hard
41*	EDA	OA	2	15.60		Yes		Yes	Hard
42	EDA	GA	na	15.51		Yes		Yes	Hard
43	BNH <sub>2</sub>	GA	na	-∞	Yes	Yes		Yes	
44	PEDG	PO	6	-	Yes				
45	PEDG	EPP	6	-	Yes				
46	PEDG	PDGE	6	-	Yes				
47	PPDG	PO	6	-	Yes				
48	PPDG	EPP	6	-	Yes				
49	PPDG	PDGE	6	-	Yes				

+ film 12 resulted in polymerization of the intercalator solution on addition of the acrolein

\* considerable precipitation of intercalator/cross-linker complex

∞ although film 43 is stable no d-spacing was measured due to the non-homogeneous nature of the treated film



## CLAIMS

1. A method of stabilizing a clayey geological formation  
5 surrounding a hydrocarbon well comprising the step of  
treating the formation with a first reactant and a second  
reactant, characterized in that a reaction of said first  
and said second reactant is essentially initiated in the  
presence of said clayey material.  
10
2. The method of claim 1, wherein the reaction is  
characterized by substrate intercalation or condensation  
stabilization with or without pH adjustment or  
15 stabilization through epoxide ring opening under neutral or  
acidic conditions or substrate in-situ polymerization or a  
combination thereof.
3. The method of claim 2, wherein the first reactant is a  
20 diamine or a polyhydric alcohol and the second reactant  
comprises at least one carbonyl group.
4. The method of claim 2, wherein the first reactant is a  
25 diamine or a dihydric alcohol and the second reactant  
comprises at least one carbonyl group.

ABSTRACT

5 A method of stabilizing a clayey geological formation  
surrounding a hydrocarbon well is described. It comprises the  
step of treating the formation with a first reactant and a  
second reactant, characterized in that a reaction of said first  
and said second reactant is essentially initiated in the  
10 presence of clayey material. The reaction can be characterized  
by substrate intercalation or condensation stabilization with no  
pH adjustment or stabilization through epoxide ring opening  
under neutral or acidic conditions or substrate in-situ  
polymerization or a combination thereof.

15



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